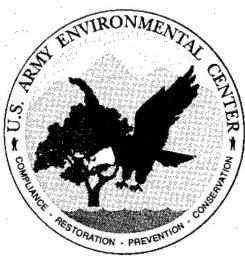


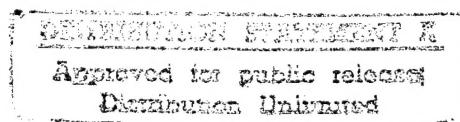
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**U.S. Army
Environmental
Center**

Composting of Nitrocellulose Fines Regulatory and Logistical Feasibility BAAP Installation Position Paper

Report No. SFIM-AEC-ET-CR-96151
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<p>13. ABSTRACT (Maximum 200 words) The production of nitrocellulose for munitions purposes results in the production of nitrocellulose fines (NC fines). BAAP currently has stored approximately 500 tons of NC fines (dry basis). Composting has been evaluated as a means of managing these fines and yielding a nonreactive beneficial soil amendment.</p> <p>This paper summarizes the regulatory logistical and feasibility of the following end-use options for the finished NC fines compost: (1) land application (with harvesting) by the installation; (2) providing local farmers with compost as a soil amendment; (3) application for mining reclamation; and (4) disposal of the compost in a landfill. Mining reclamation was not logically feasible. All of the end-use options were found to be feasible with regard to regulatory constraints. Because finished NC compost is not specified in federal or Wisconsin state regulations for solid waste, the nonhazardous nature of the compost needs to be assured through demonstration of nonreactivity and/or chemical content determination. Based on predicted compost application rates, the anticipated 450 tons/year of compost would require approximately 320 acres/year of land. Based on preliminary site selection criteria, these land requirements appear to be achievable. Total costs per ton of compost (including disposal) range from \$3,030/ton for supply to local farmers to \$3,250/ton for application by the installation.</p>			
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DRAFT NC FINES COMPOSTING POSITION PAPER
BADGER ARMY AMMUNITION PLANT

INTRODUCTION

The production of nitrocellulose (NC) for munitions purposes also results in the formation of nitrocellulose fines (NC fines). The U.S. Army Environmental Center (USAEC) is evaluating methods to recover these NC fines or to recycle them into usable products. One technology, which the USAEC has considered for NC fines or residual NC fines in soil, is biological treatment via composting.

NC is a highly substituted cellulose fiber, which is synthesized from cellulosic materials such as wood pulp or cotton, by nitration using nitric and sulfuric acids, followed by various additional processing steps.^(1,2) NC is used by the Army as a propellant (alone or in combination with other constituents) in munitions and rocket motors.

The manufacture of NC results in the production of NC fines, which are difficult to recover during production due to their small size. These NC fines have historically been discharged with process water into lagoons. Fines that settled in the lagoons were periodically removed for recycle into product or placed into storage.

While NC fines are not considered toxic by the U.S. Environmental Protection Agency (EPA)⁽³⁾, they may be reactive under certain conditions. The Army is investigating options to maximize both the recovery of the NC fines and the recycling of NC fines into usable products.⁽⁴⁾ The USAEC is evaluating composting as a method for treating NC fines, which have not or cannot be effectively recovered or recycled into propellant products. Previous testing by the USAEC has shown that composting can treat NC fines in soil.⁽⁵⁾

Badger Army Ammunition Plant (BAAP) is located in Sauk County, Wisconsin. Although the plant's production facilities are not currently active, approximately 500 tons of NC fines are being stored at BAAP. It is anticipated that these NC fines could be treated by composting to yield a nonreactive soil amendment suitable for beneficial use. Testing has shown that a compost matrix containing approximately 10 to 35% NC by weight, with a moisture content of 30%, may be handled safely.⁽⁵⁾

Several methods of composting, derived from solid waste composting practice, may be applicable for NC Fines. Based upon USAEC's work to date, it is likely that NC fines would be composted on site, using the windrow composting method. NC fines would be mixed with organic amendments which serve to support the composting process. As with other types of explosives/propellant composting, the basic process would consist of the following principal steps:

1. NC fines receipt from storage.
2. Organic amendment receipt and preparation.
3. Windrow construction.
4. Windrow operation.
5. Windrow removal and disposition of finished compost.

The general windrow composting procedure to be used is depicted in Figure 1. Amendments would be layered in long, parallel rows ("windrows"). In this approach, NC fines would be transported to the composting pad from storage. The NC fines would be placed on the amendments and mixed with a windrow turner. The windrows would be periodically turned and monitored during the process.⁽⁵⁾ At the end of the required composting period, the finished compost would be removed from the pad for use as a beneficial soil amendment.

REGULATORY REQUIREMENTS FOR COMPOST USE

A federal and state (Wisconsin) regulatory review was conducted to determine regulations and technical requirements that may affect management and disposal options for NC compost. The use of NC fines compost as a soil amendment, or for similar uses, may require demonstration that NC fines are not RCRA Listed or Characteristic hazardous wastes.

The Army has successfully held that NC fines resulting from their production processes are not RCRA Listed wastes. NC fines do not exhibit the RCRA characteristics of ignitability, corrosivity, or toxicity. Although NC fines can be reactive under specific conditions, particularly when dry, the finished compost mixtures will not be reactive because NC fines will have been treated to levels determined to be nonreactive in tests conducted by Radford Army Ammunition Plant (RAAP) for USAEC. In particular, final NC concentrations will be less than 10% NC. Results of the preliminary tests by RAAP for USAEC indicated that at NC concentrations less than 12%, NC fines compost was nonreactive at all moisture levels. Results of the RAAP Analysis were summarized in the "Composting of Nitrocellulose Fines-Hazards Analysis" report.⁽⁵⁾

It may be necessary to confirm that the finished NC compost is not hazardous and does not exhibit the RCRA Characteristic of reactivity prior to placement of the finished product on land. Reactivity tests such as the Bureau of Mines procedures used in the explosives industry may be used in directly assessing reactivity. Federal regulations describing the properties of solid wastes that exhibit this characteristic are found in 40 CFR 261.23. However, reactivity testing as a routine monitoring procedure may be expensive and time consuming. Alternatively, it may be possible to use chemical analysis methods to confirm that the final NC levels are below the established reactive level.

Review and comparison of the state (WI) and federal regulations indicates that their criteria for identifying hazardous wastes are equivalent. Therefore, a solid waste

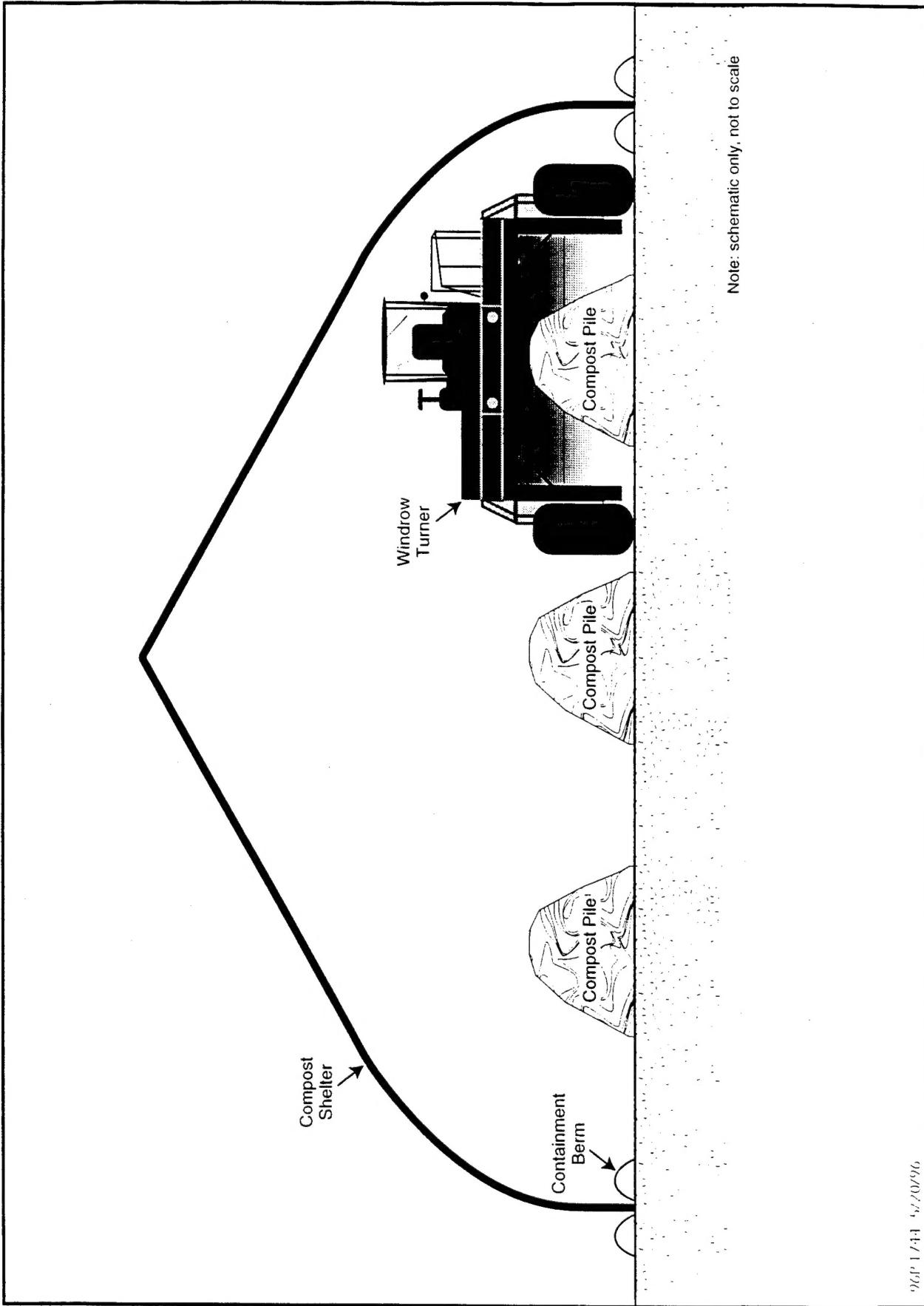


FIGURE 1 CROSS-SECTIONAL SCHEMATIC OF WINDROW COMPOSTING

considered nonhazardous under the federal regulations would be considered the same under Wisconsin regulations. Since it is expected that the finished compost will be considered a nonhazardous solid waste under federal regulations, it is also expected to be considered the same under the state regulations.

Evaluation of Potential End-Use Options:

Hazardous and solid waste regulations were examined with respect to specific applications of the finished compost. These applications may include:

- Land application (with crop harvesting) by the installation.
- Providing local farmers with compost as a soil amendment.
- Landfilling the finished NC fines compost.

Land application of compost to aid in reclamation of land disturbed by mining operations is also a usable alternative in many cases. However, surface mining operations do not exist in the vicinity of BAAP. Therefore, land application of compost for mining reclamation was not considered. The following paragraphs contain a discussion of applicable or potentially applicable regulations for these end-use options.

Land Application (by installation or by farmers):

Wisconsin regulations for land spreading of solid waste were considered to be potentially applicable for land application of finished compost. These regulations would generally apply whether the material is applied on-site by the installation or distributed to local farmers. Based on these regulations, it is anticipated that the finished compost materials must meet the following regulatory criteria to be acceptable for land application:

- A solid waste land spreading plan must be submitted.
- A waste characterization is required.
- Land spreading and operation requirements must be met.
- The compost must meet allowable concentration limits for heavy metals.

The State of Wisconsin has promulgated very specific regulations concerning the land application of solid wastes. However, there do not appear to be any insurmountable impediments for use of this disposal method for the finished NC compost. Therefore, land application of finished NC compost on-site or by distribution to local farmers can be a viable means of disposal, from the regulatory standpoint.

Landfilling:

The Wisconsin regulations for landfilling of nonhazardous solid waste do not contain any special provisions for finished compost materials. However, the Wisconsin Administrative Code states that only waste types and sources listed in the landfill's plan of operation may be accepted for disposal. If the finished NC compost is not considered as one of the

acceptable waste types or sources, its acceptance would have to be approved by the Department of Natural Resources. It appears that the nonhazardous NC finished compost can meet sanitary landfill requirements in the State of Wisconsin. Although there may be some additional data requirements, landfilling can be a viable means of finished compost disposal.

TECHNICAL ISSUES TO BE ADDRESSED

Based on an analysis of end-use options, it appears that composting is a technically feasible approach for management of NC fines once their potential reactivity has been reduced.

Prior to implementation of an NC fines composting process, there are four key technical issues that must be addressed. These issues are:

- Measurement of reactivity.
- Compostability of NC fines.
- Safety during treatment.
- Economics and reuse of NC fines compost.

These issues will be discussed individually.

Measurement of Reactivity:

As noted previously, NC fines do not pose an environmental concern due to toxicity, but only due to their potential reactivity. This suggests that treatment to destroy the reactive characteristic would render these materials environmentally safe. Therefore, the first key technical issue to be addressed involves measurement of NC fines reactivity. Due to the reactive nature of NC fines, particularly when dry, an accurate assessment of NC fines and NC fines compost is needed. As noted above, reactivity of NC fines compost could be evaluated either by direct reactivity testing or by NC analysis and correlation of residual NC levels to reactivity.

Radford Army Ammunition Plant (RAAP) performed reactivity testing on NC fines compost mixtures for USAEC.⁽⁶⁾ Testing was conducted using NC alone and in combination with three compost mixtures at various moisture levels. Reactivity to flame and shock stimuli were evaluated using standard test procedures to determine the maximum NC levels and minimum moisture levels necessary to control reactivity. Additional testing for sensitivity to impact, friction, and electrostatic discharge was conducted on selected formulations for use in evaluation of potential risks during actual composting operations.

The RAAP study concluded that NC compost mixtures exhibited similar reactivity under flame and shock stimuli at moisture levels up to 25%. Above this moisture level, NC compost required more moisture to avoid flame reactivity than shock reactivity. In other

words, at a given moisture level, NC compost was more sensitive to flame stimulus than shock. NC alone (no compost) required 55% moisture to be nonreactive in the Deflagration to Detonation Transition (DDT) test.

The reactivity testing program results also indicated that dry NC compost mixtures did not react under the DDT test at less than 12% NC. Dry NC compost mixtures containing less than 10% NC did not propagate an induced explosive reaction in a <2.5-inch diameter, schedule 40 steel pipe in the Critical Diameter for Explosive Shock Propagation (C_d) test. Based on these initial findings, it appears that NC fines composting can be accomplished under conditions such that the compost mixture is nonreactive.

Reactivity testing, such as the C_d test and the DDT test, may be a suitable way of confirming that final NC fines compost materials are nonreactive prior to their disposition as a soil amendment. Reactivity testing could also be used to confirm the safety of initial mixtures prior to composting. The precise tests to be used for these purposes have not been determined at this time. In addition, the cost for such testing if required on a frequent basis, may be of concern.

Another potential method of evaluating the reactivity of NC fines compost mixtures would be to correlate the measurable NC concentration in the compost to reactivity. After a correlation is established, only analytical results would be needed, thereby eliminating costly routine reactivity testing. The use of this approach requires an accurate, reliable, and cost effective method for analysis of NC in the compost matrix. As with most environmental analysis methods, analysis of NC in compost requires the quantitative extraction of NC from the matrix and the accurate quantification of the NC in the extract, with minimal interferences from other components.

Previous NC composting studies utilized USATHAMA Method LY02, modified for the extraction and analysis of compost. Since this method analyzes for the nitrite ion, other extractable nitrogenous compounds naturally occurring in the amendment ingredients may affect its accuracy.

The existing USATHAMA method uses an acetone extraction, followed by colorimetric measurement of a dye produced by reaction of the nitrite cleaved from the NC. While a methanol washing step is used to remove other sources of nitrite and prevent interference, the possible presence of alcohol-insoluble nitrogenous compounds may still be of concern. NC mixed in soil or humic material may not be completely extracted with acetone. Therefore, the difficulties in acetone extraction in conjunction with the potential nitrogenous interferences make the analytical determination of NC content by this method uncertain.⁽²⁵⁾

A preliminary literature search identified several other classes of analytical methods which, with suitable development, may hold some promise for analysis of NC. Based on current information, the most promising techniques include Size Exclusion Chromatography/Gel Permeation Chromatography⁽⁷⁾, and Size Exclusion Chromatography followed by

Electrochemical Detection.⁽⁸⁾ Further development of such procedures may be warranted in order to provide a reliable method for analysis of NC in compost.

Compostability of NC Fines

The second key technical issue to be addressed is the compostability of NC fines. The evaluation of any biological waste treatment process must address the degree to which the target components can be either biodegraded to mineral products, or biotransformed to environmentally innocuous or acceptable products. In this regard, several sources have concluded that NC is resistant to direct microbial attack, even by cellulolytic organisms.^(9,10) According to various reports^(11,12,13,14,15,16) as cited in Wendt and Kaplan⁽⁹⁾, substituted celluloses are also generally resistant to microbial attack, with the degree of resistance increasing with the degree of substitution.

Duran et.al.⁽¹⁷⁾ tested the anaerobic degradation of NC and found that, although some evidence of toxicity was observed, microorganisms derived from an anaerobic digester would degrade relatively high concentrations of NC (up to 54,000 mg/l) with appropriate acclimation. The presence of co-substrates, including cellulose, appeared to suppress the toxicity of NC. Other anaerobic degradation tests by Hsieh and Tai⁽¹⁸⁾ used acclimated microorganisms. Various enzymatic inducing agents were evaluated for their ability to foster NC degradation. The authors concluded that the tested reagents were not effective in inducing NC degradation in the test system.⁽¹⁸⁾

However, microbial transformation of NC may be possible under certain conditions. Kaplan also reports that NC is not directly metabolized by microorganisms and suggests that other studies, in which growth of microorganisms on NC was reported, may represent cases where the observed growth was on other contaminants, on unsubstituted cellulose, or due to the effects of secondary metabolites on NC structure⁽¹⁶⁾. Wendt and Kaplan⁽⁹⁾ also cite Urbanski's⁽¹⁹⁾ conclusions that microorganisms growing on other substrates may produce metabolic products that adversely affect the stability of NC. Brodman and Devine⁽²⁰⁾ report that a fungus indirectly utilized nitrogen from NC (by a hydrolysis reaction) when supplied with a supplemental carbon source without attacking the cellulose (carbon) backbone. Recent studies conducted using three fungal strains concluded that none of these organisms used NC as a source of carbon under the conditions tested.⁽²¹⁾ This suggests that although the NC was biologically transformed, the carbon backbone was not attacked.

USAEC has previously conducted tests of composting of NC in soils, primarily as a potential remedial technology for sites with residual NC in soils from previous manufacturing activities.^(22,24) The first USAEC study used both laboratory and pilot scale testing to evaluate whether NC in soils from Badger Army Ammunition Plant (BAAP) in Baraboo, Wisconsin, could be metabolized by composting.⁽²⁴⁾ In addition to residual NC soil trials, radiolabelled NC (uniformly labeled ¹⁴C-NC) was added to soil used in test composts (in addition to the NC contributed by the contaminated soil) in order to assess the fate of NC in the compost.⁽²⁴⁾ The evolution of ¹⁴CO₂ from radiolabelled substrates is

generally taken as evidence of mineralization; alternatively, the ^{14}C tracer may allow analysis of the partitioning of the organic compound within the matrix. In contrast to previously cited sources, results of the BAAP testing indicated that, under simulated composting conditions, biodegradation of the cellulose backbone occurred. Subsequent pilot-scale testing at BAAP indicated that NC degradation well in excess of that attributable to thermal degradation was seen to occur.

Finally, a field-scale demonstration was conducted at BAAP to evaluate the potential utility of aerated static pile composting as a treatment and remediation technology for NC fines and residual NC fines in soil.⁽²²⁾ Results of this field demonstration indicated that composting is a feasible technology for reducing the extractable NC concentration in contaminated soils. In addition, this field demonstration provided evidence that NC at a high concentration can be degraded when incorporated into a compost mixture⁽²²⁾, using small amounts of compost spiked with high concentrations of pure NC, incubated in porous bags, placed within some of the test piles.

Although mineralization (i.e., conversion of organic components to carbon dioxide and mineral products) is often the optimal treatment result, it may not be strictly necessary in all cases. Rather, treatment to render the material nonhazardous may be acceptable. As noted previously, NC is not toxic. Thus a process that transforms NC into nonreactive material may meet the primary treatment goal.

Safety During Treatment:

The third technical issue that merits attention is safety during treatment. Due to the energetic nature of propellants, which can result in detonation under shock or thermal stimuli, safety criteria and procedures to avoid these stimuli are of critical importance in all materials handling aspects of their treatment. Safety criteria must consider the levels of propellant that can safely be handled in the treatment process. NC is known to be a potentially reactive material, particularly when dry. As previously stated, results of the RAAP Hazards Analysis indicated that dry NC compost mixtures did not exhibit reactivity at less than 12% NC. It was demonstrated that NC fines at loading rates less than 35% in a compost matrix, with a moisture content of 30%, may be handled safely. Therefore, it is anticipated that composting operations could be safely conducted if starting compost mixtures were maintained below 35%, with moisture maintained at or above 30%. Treatment would be continued until NC concentrations were less than 10%, at which point the material would be nonreactive regardless of moisture content.

In addition, an explosives hazards safety analysis would be conducted on the selected compost materials handling and operating equipment prior to their use. This analysis would consider the potential initiating forces which might be produced by the machinery, relative to the reactivity of the material. Only equipment deemed safe on the basis of such a review would be approved for use in the NC fines composting operation.

Economics and Reuse of NC Fines Compost:

The last technical issue to be addressed involves the economics and reuse of NC fines compost. The economics of the composting process itself were discussed in "Composting of Nitrocellulose Fines- Hazards Analysis".⁽⁵⁾ Potential end-use options for the compost were evaluated based on logistical and economic factors discussed in "Composting of Nitrocellulose Fines - Regulatory and Logistical Feasibility, BAAP Installation."⁽²³⁾

Project costs for the NC fines composting facility were estimated using a NC fines throughput of approximately 500,000 lb/year (on a dry basis), and a 35% NC fines loading at 30% moisture. This would allow treatment of the 500 tons of stockpiled NC at BAAP in approximately 2 years of operation. It is anticipated that the nonreactive soil amendment product of the composting process would be suitable for beneficial reuse. The follow end-use options have been evaluated for BAAP:

- Land application (with crop harvesting by the installation).
- Providing local farmers finished with compost as a soil amendment.
- Disposal of finished compost in a landfill.

All of the end-use options were found to be feasible with regard to regulatory constraints. To apply the anticipated 450 tons/year of finished compost, approximately 320 acres/year of land will be needed, based on predicted application rates. Table 1 shows approximate annual costs for each end-use option⁽²³⁾.

Based on these analyses, it is projected that composting could be implemented as a management method for NC fines at BAAP at a total cost ranging from \$3,030 to \$3,250/ton (dry basis), depending on the end use option. Total costs are presented in Table 2.

RECOMMENDATIONS

USAEC has previously tested composting of NC in soils, primarily as a potential remedial technology for sites with residual NC in soils from previous manufacturing activities. Results of these studies, in conjunction with findings from a preliminary literature review, indicate that NC fines may be treatable via composting. The composting process is anticipated to yield a nonreactive soil amendment suitable for beneficial use. For further development of composting as a treatment technology for NC fines, the following factors should be addressed:

- Determining the composting period and operating conditions necessary to achieve the desired NC destruction, and corresponding reactivity reduction.

Table 1

Annual Cost Summary of End-Use Alternatives for NC Fines Compost

Alternative	Estimated Annual Cost (\$/year)	Estimated Cost per Ton of Finished Compost (\$/ton) ^a	Estimated Cost per Ton of Original NC Fines (\$/ton) ^b
Land Application by the Installation	59,400	90	260
Supply to Local Farmers for Land Application	8,300	20	40
Compost Landfilling	40,500	65	180

^aBased on 640 tons/year of compost, wet basis.

^bBased on 250 tons/year of NC fines, dry basis.

Table 2

**Total Compost Cost
(2 Year Project)**

Alternative	Compost Process (\$/ton NC Fines)	Compost End Use (\$/ton NC Fines)	Total (\$/ton NC Fines)
Land application by the installation	3,000	250	3,250
Supply to local farmers for land application	3,000	30	3,030
Compost landfilling	3,000	170	3,170

- Evaluating the need for adding additional carbon sources (amendment supplementation) after temperatures have dropped in the windrow to prolong the composting period and achieve additional NC degradation, if needed.
- Maintaining environmental parameters (i.e., moisture, temperature, pH, and oxygen) such that transformation of contaminants is optimized and the potential for reactivity of NC fines is minimized.
- Determining safe materials handling procedures for initial NC fines/compost mixing and compost pile maintenance.
- Confirmation of a correlation between measurable NC levels and reactivity.
- Evaluating the current analytical method versus potential new analytical methods for NC fines compost.

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